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Research Article

CHARACTERIZATION OF FOUR BALL METALLIC WEAR MECHANISMS USING SCANNING ELECTRON MICROSCOPY

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ABSTRACT:
Nowadays, lubricants play an important role in several automotive industries around the world because they reduce friction and wear on engines moving parts such as piston ring, cylinder liner and valve control systems of compression ignition engines. The aim of this research was to investigate the impact of bio-oil (palm oil) on metallic wear. The tribological test was conducted using a four-ball tribometer as indicated by engineering testing standard ASTM (D4172) under the conditions of 392 N applied load, 75°C and the period of 60 minutes. These four ball surfaces were observed by using 3D Optical Microscope (OM) and Scanning Electron Microscope (SEM) analysis. According to the four-ball wear test, the comparison of average wear scars diameter between two different types of bio-oil and SAE 0W30 engine oil were investigated. Furthermore, wear scar depth from 3D microscope and SEM images of different magnification were compared in the viewpoint of wear mechanism analysis.

Keywords: *Four-ball Wear Tester, Scanning Electron Microscopy, Wear Mechanism*

1. INTRODUCTION

Lubrication is one of the most critical parameters of an internal combustion engine, since it helps to keep it running smoothly and to prevent engine deterioration. Lube oil serves a variety of purposes, including cooling, friction reduction, and wear control. Between moving surfaces, the lubrication oil forms a lubricating layer, which lowers friction and wear.

Petroleum-based lubricants, such as mineral oil, are widely used in industrial lubrication, particularly in the mechanical and engine industries. However, because mineral oil-based lubricants are not readily biodegradable and hazardous, their use poses a risk to our environment, perhaps causing a greenhouse effect and contributing to global warming. Vegetable oil as a lubricant in the lubrication industry is not a new discovery, coconut oil was utilized as a lubricant for two-stroke engines. There are numerous advantages of replacing mineral oil with vegetable oil as lubricants, including the fact that vegetable oil is biodegradable, non-toxic, renewable, and affordable. Vegetable oil's other advantages, such as its high viscosity index, superior lubricity, high flash point, and minimal evaporative loss, have made it a better alternative to mineral oil-based lubricants. As a result, as global concerns about the production of environmentally friendly lubricants grow, vegetable oil-based lubricants have begun to replace mineral oil-based lubricants in the lubrication sector. Palm oil is a vegetable oil-based lubricant that has been extensively tested in the lubrication business due to its advantages over mineral oil, including its low cost, easy availability, biodegradability,

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environmental friendliness, and renewable nature. The tribological properties, such as anti-wear, anti- friction, viscosity index and flash parameter point of RBD palm olein with mineral oil in different blending volume ratio are investigated [1].

In comparison to mineral-based oils, vegetable oils are a renewable supply that is easy to replicate. Vegetable oil has a relatively low volatility due to the high molecular weight of its triglyceride molecule. Palm kernel oil (PKO), which has a semi solid phase, is mixed with varying weightage percentages of poor point depressant (PPD) as a bio lubricant to examine low temperature behavior and to assess the influence of lubricity performance when blended with different percentages of PPD (5wt percent, 10wt percent, 20wt percent and 30wt percent). It is reported that by adding PPD it can significantly improve the lubricity performance in terms of surface roughness [2]. Additives extend the life of the lubricant and provide additional performance features such as enhanced flow, reduced friction, oxidation resistance, and pressure or temperature stability. It is observed that the presence of the boundary lubrication regime is indicated by the friction behavior of Palm Oil Methyl Ester (POME) as an addition in commercial lubricant. According to the results of the viscosity tests, 5% POME can increase the viscosity index VI qualities of mineral-based lubricants up to 500 N load. [3]. Palm oil-based TMP (trimethylolpropane) ester is made from palm oil, which is biodegradable and has excellent lubricity qualities like a higher flash point temperature and VI (viscosity index). The lubrication regimes of hydrodynamic, elasto-hydrodynamic, and boundary lubrication are all examined in order to evaluate wear prevention characteristics in this study [4]. However, The poor oxidative and thermal stability of vegetable oil leads to the formation of sludge, deposits and corrosive in metal surface.

Antioxidants can prevent oxidation by protecting the lubricant from oxidative deterioration, providing those industrial specifications are followed. It is presented that involved combining curcumin-extracted soybean waste cooking oil in three distinct compositions (10%, 20%, and 30% v/v) with the mineral base oil N-150 to create WCO-1, WCO-2, and WCO-3 Biolubricant formulations. Whereas, tribological analysis demonstrates that wear scar diameter has significantly reduced from 0.685 to 0.573 mm, and the coefficient of friction decreased from 0.117 to 0.080 with respect to the mineral base oil. Although the waste cooking oil's low oxidative stability, natural antioxidant curcumin was added to compensate for the thermal and oxidation stability [5]. Sometimes, the engine oil become impurities of contamination during serves as lubricating functions. They are in the form of nanostructure of solid, liquid and gaseous contaminants. One of the research works has been found that the different conditions of soot contamination in engine oil under four ball tests with four types of carbon black or without carbon black at different weight ratio. It is recommended that the primary particle size of carbon black N330 is similar to the size of the soot particle from diesel engine exhaust (29-31 nm) which is smaller than the calculated oil film thickness [6].

The area of grooves, plastic deformation and subsurface crack can be analyzed SEM micrograph of metallic wear scar for the engine oil without soot. When carbon black is added to the oil, however, it is visible that there are many deep grooves along with the sliding direction. Metallic wear mechanisms are clearly explained by the relationship between calculated oil layer thickness, primary nanoparticle size distribution, carbon atom density of soot, and hardness [7]. Because unburned biodiesel directly impacts on the cold walls of the combustion chamber and is swept to the oil pan, engine oil dilution occurs, causing changes in oil friction, wear, and lubricity qualities. This study can be proposed that when compared to the other studied mixes, the oil contaminated with animal fat feedstock (e.g., chicken fat) biodiesel demonstrated the best wear behavior (of mineral oil with vegetable feedstock biodiesel dilutions) [8].

In the previous research, even though most of researchers have investigated in wear performance, friction, surface roughness, oil firm thickness and lubricity properties of engine lubrication sectors by replacing biodegradable lube oil, there are less still presented in 3D micrograph of bio lubricant and new synthetic lubricant 0W-30. Therefore, this work focusses on the experimental observation of wear performance and wear scar, analysis of the micrograph and depth scars of pure palm oil Biolubricant and SAE 0W-30 by using Four Ball Tribology Test, Scanning Electron Microscopy (SEM) and 3D Optical Microscope. It can be known that the differences of wear scars parameters, friction torque and deep scars between bio lube oil and synthetic new lubricant 0W-30 under the observation.

2. MATERIALS AND METHODS

Natural oils present an attractive substitute to conventional lubricants, especially in environmentally sensitive areas like agriculture, forestry, and mining since they have low toxicity, high biodegradability low friction and wear characteristics, and improve the surface finish. However, natural triglycerides have some drawbacks, including a low flash point, and poor thermal stability.

2.1 Biolubricant and Synthetic lubricant

In this research used palm oil to be the representative of Biolubricant by compared with synthetic SAE 0w-30 lubricating oil in order to identify wear performance and wear scar. In order to investigate the wear performance and wear scar of Biolubricant, this experiment uses SAE 0W-30. The specification of sample, equipment and condition for wear test were shown as following. Sample of bio-oil and new oil (OW-30) can be described in Fig. 1.

Table 1 shows different viscosity at 40°C and percent of carbon, hydrogen and oxygen contents of Bio lubricant, new synthetic in this section test were test in four ball testers to know in case of wear and wear scar to identify the performance of wear of each sample.

Table 2 shows Palm oil properties, which are maintained based on ASTM specifications. However, producers and refiners must pay more attention to meet the requirements. One of the important requirements is a sulfur-free diesel or diesel oil with 0.05% sulfur content, which is very expensive. With these considerations, many studies have searched for an alternative lube from renewable sources, which could substitute the conventional lubricating oil.

Table 3 shows lubricating oil specification of synthetic SAE 0w-30, viscosity is 44.5 cSt at 40°C and 9.6 cSt at 100°C and total base number (TBN) is 5.6 mg KOH/g. This synthetic lubricant is an important item in our study as it is used to estimate the wear performance in comparison with the Biolubricant oil. The performance of synthetic lubricant is an extreme property case to prevent wear scar in engine and also resistance of degradation, it is high when compare with Biolubricant.

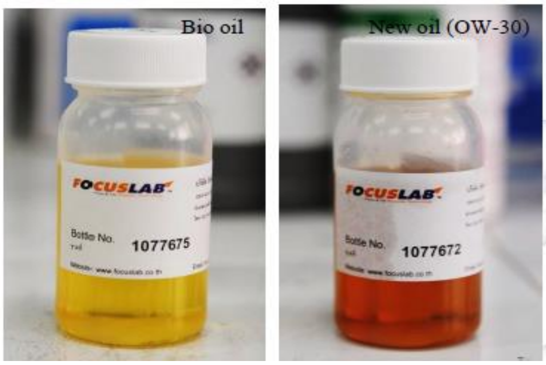

Fig. 1. Sample of Bio Oil and New Oil (0W-30).

Table 1: Ranges of parameters in the investigation.

Fuel	Viscosity at 40°C (cSt)	Carbon (%)	Hydrogen (%)	Oxygen (%)
Biobricant (Palm Oil)	4.5	0.23	34	4.5
SAE 0W-30	44.5	95	5	0

Table 2: Biolubricating oil (Palm oil) specification (Based on ASTM Standard)

Properties	Specification
Density (kg/m^3) at 15°C	918
Kinematic Viscosity (mm^2/s)	95-106
Flash point (°C)	280
Cetane number	40
Pour point (°C)	31
Sulfur (Wt%)	0.01

Table 3: Lubricating oil SAE 0w-30 specification

Properties	Specification
Viscosity @ 40 °C, (cSt)	44.5
Viscosity @ 100 °C, (cSt)	9.6
Viscosity Index	207
Oxidation	18.1
TBN, (mg KOH/g)	5.6

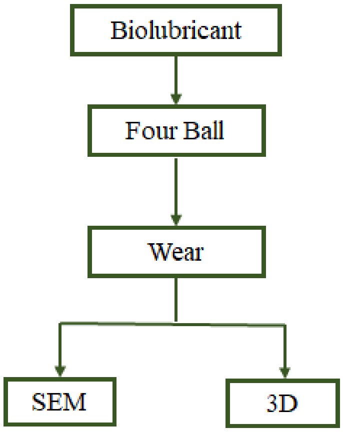

Fig. 2. Schematic diagram of methodology

2.2 Experimental procedures of Biolubricant (Methodology)

Figure 2 depicts the schematic diagram of methodology in Biolubricant and New lubricating oil which used in this research. There are two main tests the first test is used lubricating oil characteristic. In this part consist of wear performance and wear scar in optical test. Table 4 expresses the value of accuracy and uncertainty of measurement devices utilized in this study. According to the test results, the uncertainty of measurement can be seen by wear scar diameters is about ± 0.3 µm. The uncertainty of the different observed values of friction torque is ± 4 N•mm.

Table 4: Accuracy and uncertainty of measurement devices [9]

Measurement	Apparatus	Accuracy
Friction and wear	Four Ball Tester (ASTM D 4172)	-
Wear scar diameter	Optical Microscope (ASTM D 4172)	± 0.01 mm
Scanning Electron Microscope (SEM)	Scanning Electron Microscope	3.0 nm at 30 kV

2.3 Equipment of wear test

Figure 3 shows the schematic diagram of four balls to know, it is for tribology test in case of wear and friction between two types of lubrications. The Four Ball Tester can be used to determine Wear Preventive properties (WP), Extreme Pressure properties (EP) and friction behavior of lubricants. The wide acceptance of test results of the four-ball tester makes it an excellent choice to benchmark products. It is a good choice for R&D due to its relatively inexpensive samples and ability to produce quick and repeatable results.

A four-ball tester in this study enables the development and research of innovative analytical lubricants. The operation of this device is by using four balls to examine a specimen. Three balls are locked together in the fuel sample bowl and anther ball rotates above those the three balls. The 4-ball set is brought to the upper end via a set of electric motor rotating shafts at a constant speed [10]. The test load, duration, temperature, and rotational speed are set in accordance with standard test schedule. In Wear Preventive (WP) tests also called Anti Wear (AW) tests provide the average scar diameter on the bottom three balls is reported.

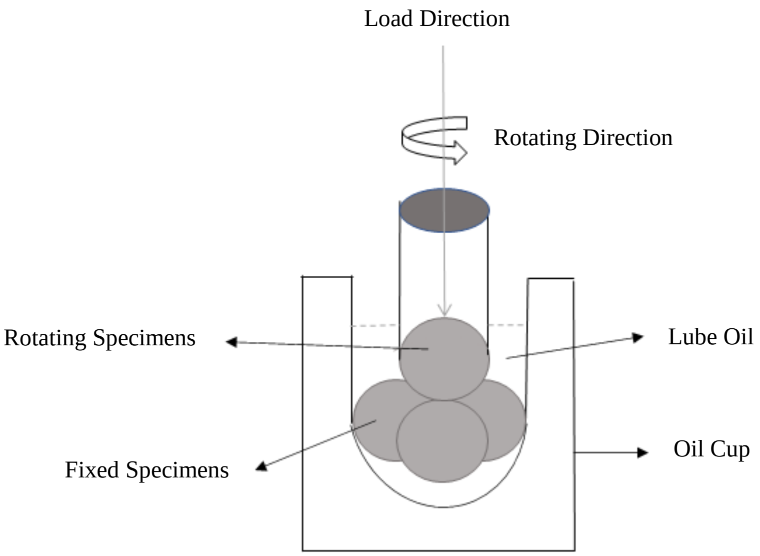

Fig. 3. Schematic diagram of four ball tester

Table 5 shows conditions of the four balls tester, which is an excellent development and quality check instrument for developers and users of lubricants and additives. The unique sample configuration of three bottom balls and one top ball makes a very stable and a repeatable contact in-turn, allowing tests results to be very repeatable. The samples are tested at 75°C. The normal applied load is 392 N at running time is 60 minutes according to the four balls testing standard (ASTM D4172).

2.4 Equipment of wear scar test

Figure 4 shows the 3D optical microscope for finding the size of wear scar that generated from four ball tester. 3D optical microscope has become a versatile tool for measuring areal surface topography and texture in a wide variety of industries. A key factor in the growth of industrial applications has been hardware and software implementations that streamline and simplify its use, particularly for technician-level users.

Table 5: Condition test of four ball tester

Parameter	Specification
Rotational speed	1,200 rpm
Load	392 N
Duration per load	3,600 Sec
Temperature	75°C

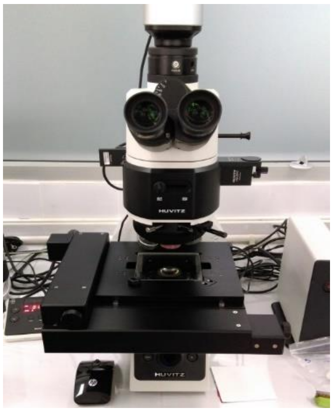

Fig. 4. 3D optical microscope

Quantitative 3-D surface metrology measuring surface topography as well as the size and shape of microscopic surface features – is vital to many industries. In applications including coated glossy paper, painted automobiles or molded plastic parts, surface texture affects cosmetic appearance and, hence, perceived value. But in many cases, such as with automobile engine components, medical implants, or high-brightness LEDs (HB-LEDs), the surface topography can be absolutely critical to proper part functioning.

Figure 5 shows Scanning electron microscopy (SEM) is a method for high-resolution imaging of surfaces. The SEM uses electrons for imaging, much as a light microscope uses visible light. The advantages of SEM over light microscopy include much higher magnification (>100,000X) and greater depth of field up to 100 times that of light microscopy. Qualitative and quantitative chemical analysis information is also obtained using an energy dispersive x-ray spectrometer (EDX) with the SEM. The SEM generates a beam of incident electrons in an electron column above the sample chamber.

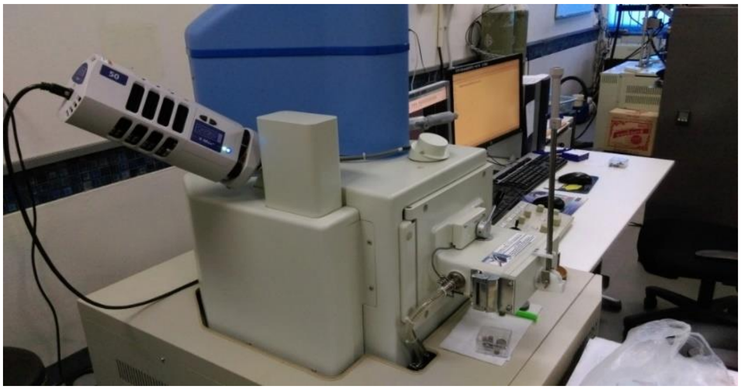

Fig. 5. Scanning electron microscopy (SEM)

3. Result and Discussion of wear

The Four-Ball testing is one of the tribological test methods. This test method can be used to determine the relative wear preventive properties of lubricating fluid in sliding contact under the prescribed test conditions. No attempt has been made to correlate this test with balls in rolling contact.

3.1 Friction Torque of Four ball tester

Winducom software was applied to measure values of the mean friction coefficient. Friction torque was calculated by Equation (1). With the load cell's usage in the instrument, the frictional torque was calculated (IP-239 standard, 1986 [11]). The same technique was also used by Zulkifli et al. [12].

$$T = \frac{\mu \times r \times 3W}{\sqrt{6}} \tag{1}$$

in which, T is frictional torque (Nm), μ is coefficient of friction, r is the distance measured between the contact surface center on the lowest balls and the axis of rotation, W is load (N). As a result, Fig. 6 shows impact of Biolubricant to friction torque. By after test 3,600 seconds of four ball test in each samples show the highest average friction torque is 11.72 N.mm. By minimum is 6.32 N.mm in pure new lubricating oil 0w-30.

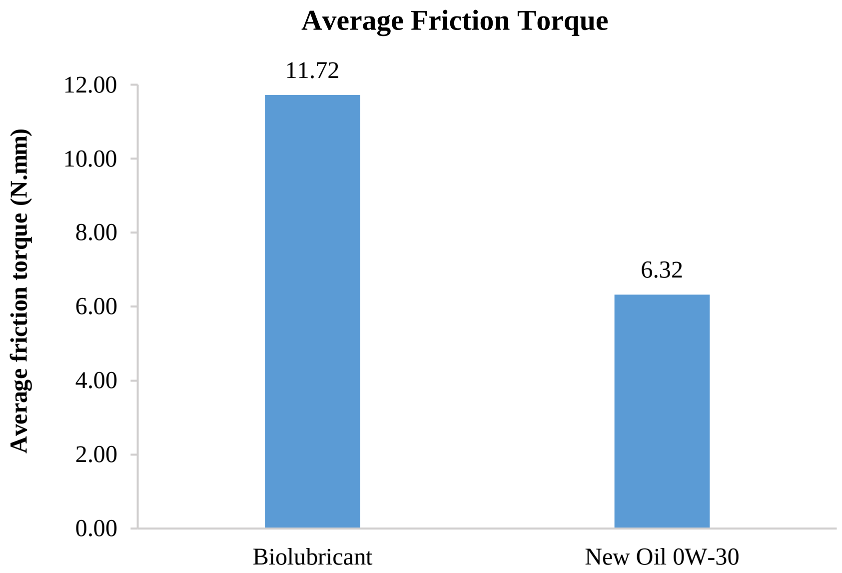

Fig. 6. Impact of Biolubricant to friction torque

Figure 7 shows friction torque by testing time to identify the individual lubrication properties of each sample. The result show that friction torque of Biolubricant is dramatically increase from 0 N.mm since 200 second to 16N.mm at 1,800 second; that is almost 2 time of new synthetic oil which is dramatically increase from 1 N.mm at 900 second to 9 N.mm at 1,260 second. This friction torque is the evidence for prove that wear performance of Biolubricant which use for lube oil in this test are poor for preventive to metal. This is possibly the molecular force of palm oil is weaker than molecular force of synthetic oil that have high resistance from shear force.

3.2 Wear scar diameter

Figure 8 shows average wear scar diameter, the result show Biolubricant has maximum wear scar diameter by wear scar is 1069.73 µm; by minimum wear scar is on pure lubricating oil is532.02 µm. The clear evidence was showed in wear scar diameter graph that wear diameter of Biolubricant is 2 time of pure lubricating oil.

This is show that if we directly used Biolubricant instead of pure new synthetic oil, in case of metal preventive it is going to be worse, since the four ball test results show Biolubricant provide poor resistance wear performance. Nevertheless, this test cannot confirm that if Biolubricant was blend with some additives which has preventive metal or improve resistance of wear performance. It might be better or worse when compare with commercial synthetic lubricating oil.

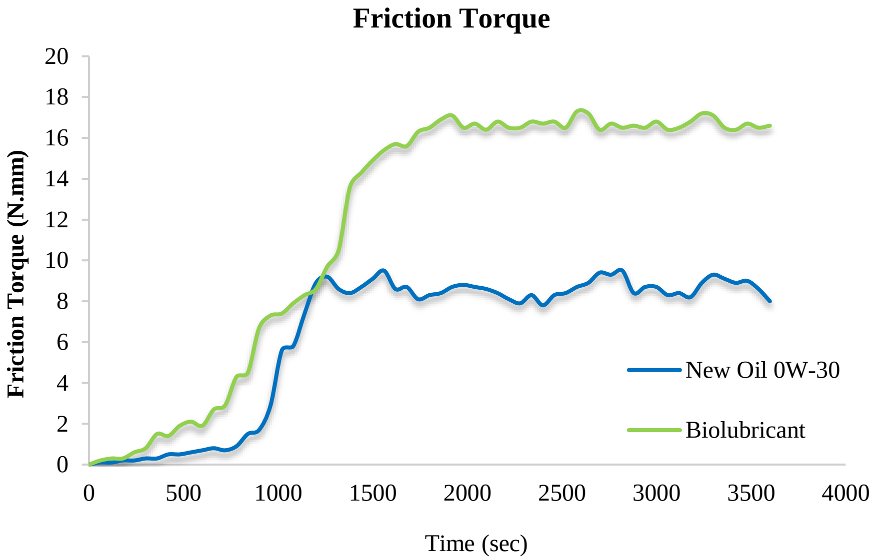

Fig. 7. Impact of Biolubricant to testing time friction torque

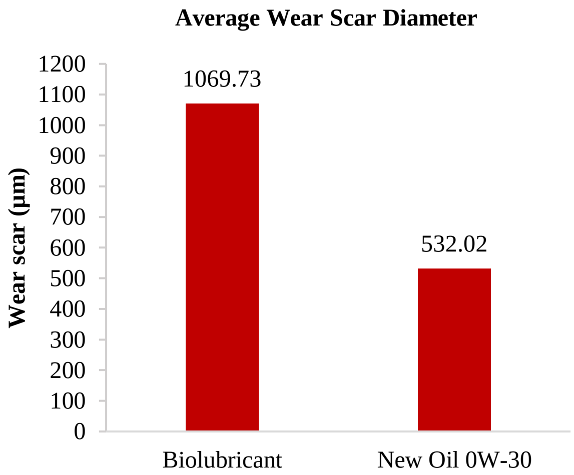

Fig. 8. Average of wear scar diameter

3.3 3D optical microscope

Figures 9 and 10 depict wear scars depth of Biolubricant and new oil 0W-30 which used for lubricating oil in four balls tester.

These Figures focus on surface analysis with 3D optical microscope. They reveal the three-dimensional shape of the lubricated wear scars by providing various width, height, and length with respect to corresponding angles. The wear scar of Biolubricant (Pure Palm oil) is big and deep, as shown in Fig. 9. On the other hand, the wear scars by the synthetic New Oil 0W-30, is relatively shallow as following in Fig. 10. According to the observations, Biolubricant deeper wear scars than commercial synthetic lubricating oil.

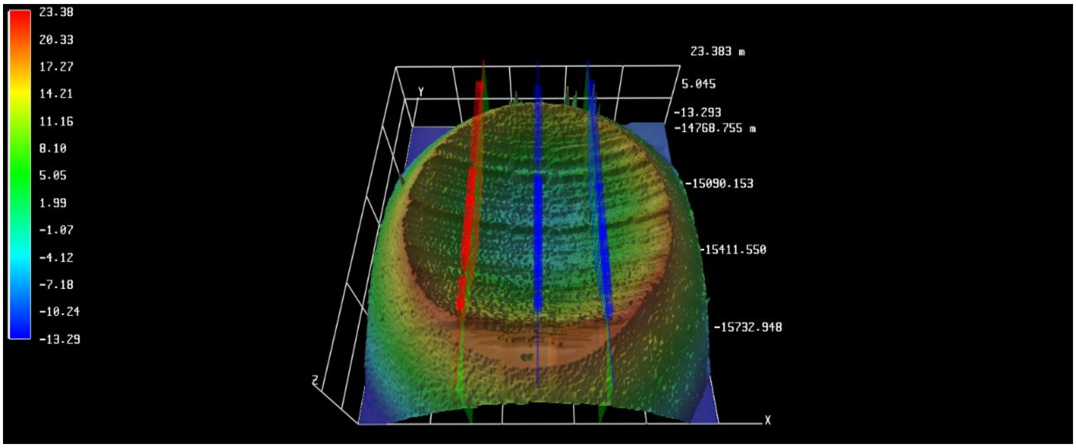

Fig. 9. Ball depth scars of Biolubricant from 3D microscope.

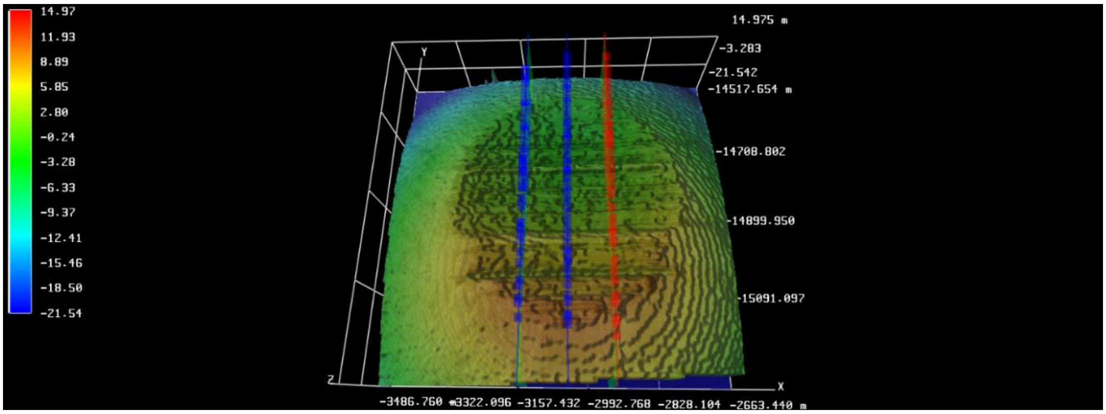

Fig. 10. Ball Depth Scars Of New Oil 0W-30 From 3D Microscope

Figure 11 shows average depth scars in each sample from 3D optical microscope. The results of depth scars can be provided by analyzing the data of height of the ball depth scars from 3D microscope. The average and maximum value of depth scar of steel ball after test from four ball tester, the results show that Biolubricant is not only has highest average depth wear scar of 2.08 µm but also has highest maximum depth scar of 5.52 µm. On the other hand, the lowest deep scar and their average value is on pure lubricating oil at 2.86 and 0.89 µm, respectively.

3.4 Wear scar from SEM image

The finding of trends and behaviors of micrographs of the area around the wear scar and the worn surfaces of the ball specimens for the different samples is similar to the findings reported by Zulkifli et al. [4]. They found wear scar diameter using palm oil based TMP ester and compared with an ordinary lubricant. Figure 12 displays the SEM images of micrograph from the stationary ball of different magnification of lubricants. This test show that wear of steel ball form four ball tribometer in case of scar from Biolubricant at the magnification of 80 times in Fig. 12(a); since wear volume is relatively bigger when compare with new oil 0W-30 ball steels. For the synthetic lubricant, 150 times magnification was applied to created image of wear as shown in Fig. 12(d). The furrows in the wear scar lubricated with 0W-30 is slightly deeper than those on the wear scars lubricated with Biolubricant. It can be investigated that even though synthetic new oil 0W-30 has smaller wear scar diameter than Biolubricant, whereas Biolubricant has better antiwear properties than commercial synthetic new oil 0W-30. Because biolubricant is characterized by much smoother with shallow furrows as compared in Fig. 12(b and e) and Fig. 12 (c and f). Therefore, synthetic 0W-30 has developed by deeper furrows and spikes, indicating that adhesive wear and fatigue deformation which were the major wear forms. By this observation, Bio lube oil has also some advantages in wear mechanism although it has some weak in friction torque, scar diameter and depth scar.

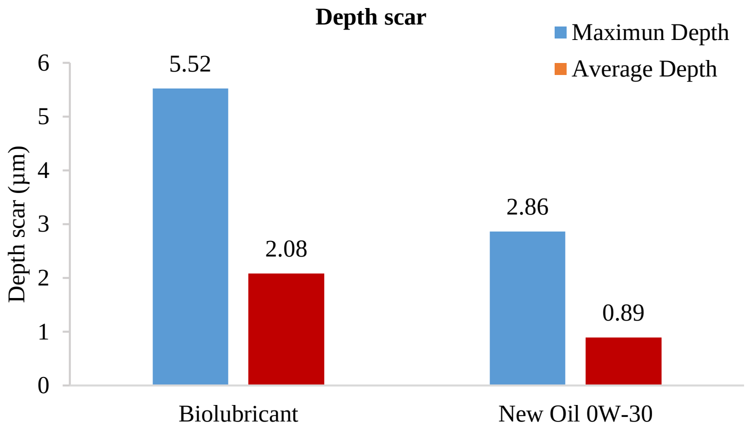

Fig. 11. Average Depth Scars in Each Sample from 3D Microscope

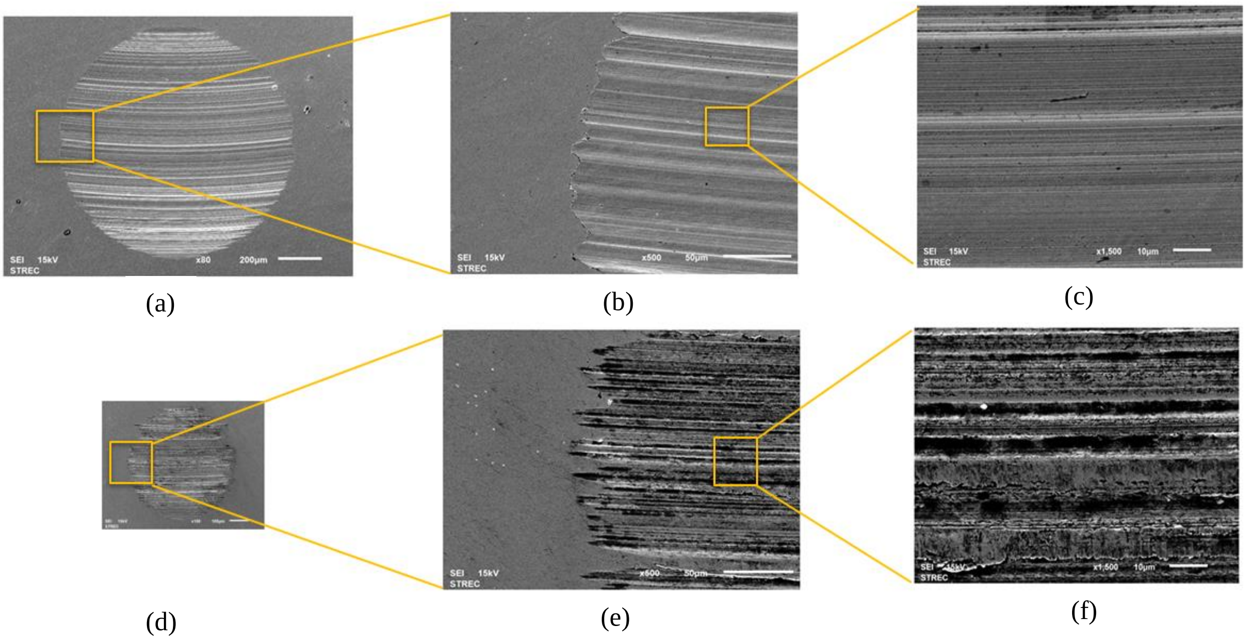

Fig. 12. SEM Micrograph from the Stationary Ball of Different Magnification of Biolube Oil and New Oil 0W-30 (a) All Bio Lube Oil_80x (b) Edge Bio Lube Oil_500x (c) Middle Bio Lube Oil_1500x (d) All New Oil 0W-30_150x (e) Edge New Oil 0W-30_500x (f) Middle New Oil 0W-30_1500x

4. Conclusion

This work focuses on friction torque and wear scar analysis used Biolubricant (Palm oil) as a lubricant. The core of this study is in the detailed analysis of SEM micrograph. New 0W-30 lubricant provides deeper furrows as compared to the Biolubricant. The main evidence is corrosive product with black colour appeared at higher temperature. This can be explained from the fact that at the higher temperature, oxidized lubricant produces different types of corrosive acids that enhance corrosive wear. This discussion can be consistent with previous research works in literature [13, 14]. Results of Biolubricant wear analysis are summarized as follows:

1. Biolubricant affects friction and wear scars of the ball by the highest friction torque is 11.72 N.mm, whereas the value for pure lubricating oil SAE 0w-30 is 6.32 N.mm

2. Although Biolubricant shows the negative effects on wear and friction, Bio lube oil has also some advantages in wear mechanism due to the high magnifications of Fig. 12(b, e) and 12(c, f) with the characterization of smoother with shallow furrows.
3. The four balls is the method to evaluate the sample oil lubricity, associated with the boundary lubrication. The result show biolubricant has poor lubricity than the synthetic oil because of highest friction torque, large and depth wear scar. Based on this result, it can be confirmed that Biolubricant (palm oil) have negative ratio in lubricity.
4. This study cannot confirm that all of Biolubricants have worse direction of lubricity performance than synthetic oil. If Biolubricant was properly blended with additives, it may result in better lubricity performance as compared pure Biolubricant utilized in this work.

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